#### CFE3320US.03/04

## DECLARATION

I, Ryuichi YAMADA, a Japanese Patent Attorney registered No. 7898 having my Business Office at Hasegawa Bldg., 4F, 7-7 Toranomon 3-chome, Minato-ku, Tokyo, Japan, solemnly and sincerely declare:

That I have a thorough knowledge of Japanese and English languages; and

That the attached pages contain a correct translation into English of the specification of the following Japanese Application:

APPLICATION DATE OF
NUMBER APPLICATION
128928/2001(Pat.) 26/APR/2001

Applicant(s)
CANON KABUSHIKI KAISHA

Signed this 20th day of Leptenber, 2005.

Ryuichi YAMADA

# PATENT OFFICE JAPANESE GOVERNMENT

This is to certify that the annexed is a true copy of the following application as filed with this Office.

APPLICATION NUMBER

**DATE OF** 

**APPLICATION** 

128928/2001(Pat.)

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Applicant(s)
CANON KABUSHIKI KAISHA

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#### [Document]

#### Specification

## [Title of the Invention]

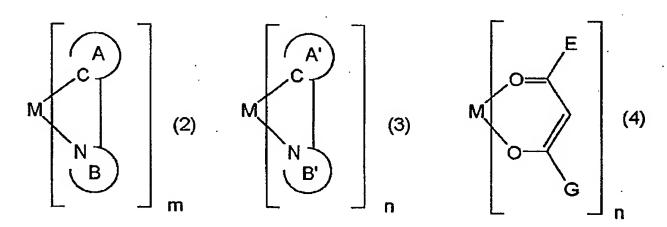
Luminescence Device and Display Apparatus

#### [Claims]

1. A luminescence device having a layer containing a metal coordination compound, which is represented by formula (1) below:

$$MLmL'n$$
 (1)

wherein M is a metal atom of Ir, Pt, Rh or Pd; L and L' are mutually different bidentate ligands; m is 1, 2 or 3 and n is 0, 1 or 2 with the proviso that m+n is 2 or 3; a partial structure MLm is represented by formula (2) shown below and a partial structure ML'n is represented by formula (3) or (4) shown below: <KA-1>



wherein N and C are independently a nitrogen atom and a carbon atom; A and A' are each cyclic group capable of having an optional substituent, including a

carbon atom and bonded to the metal atom M via the carbon atom; B and B' are each cyclic group capable of having an optional substituent, including a nitrogen atom and bonded to the metal atom M via the nitrogen atom; {(the optional substituent of the cyclic groups is selected from a halogen atom, cyano group, a nitro group, a trialkylsilyl group (of which the alkyl groups are independently a linear or branched alkyl group having 1 to 8 carbon atoms), a linear or branched alkyl group having 1 to 20 carbon atoms (of which the alkyl group can include one or nonneighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CHor -C≡C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom); or an aromatic group capable of having an optional substituent that is a halogen atom, a cyano atom, a nitro atom, a linear or branched alkyl group having 1 to 20 carbon atoms (of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C $\equiv$ C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom)};

the cyclic groups A and B are bonded to each other via a covalent bond and the cyclic group A' and B' are bonded to each other via a covalent bond;

branched alkyl group having 1 to 20 carbon atoms of which the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom, or an aromatic cyclic group capable of having an optional substituent (that is a halogen atom, a cyano atom, a nitro atom, a trialkylsilyl group of which the alkyl groups are independently a linear or branched alkyl group having 1 - 8 carbon atoms, a linear or branched alkyl group having 1 to 20 carbon atoms of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C=C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom; and

at least one of the optional substituents of the cyclic groups, and the cyclic groups B and B' include an aromatic cyclic group capable of having an optional substituent represented by the following formula (5):

<KA-2>

the optional substituent of the aromatic cyclic group is selected from a halogen atom, cyano group, a nitro group, a trialkylsilyl group of which the alkyl groups

are independently a linear or branched alkyl group having 1 to 8 carbon atoms, a linear or branched alkyl group having 1 to 20 carbon atoms of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C $\equiv$ C-; and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom, or an aromatic group capable of having an optional substituent (that is a halogen atom, a cyano atom, a nitro atom, a linear or branched alkyl group having 1 to 20 carbon atoms of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C $\equiv$ C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom) with the proviso that one or two CH groups comprising the aromatic cyclic group capable of having a substituent in the formula (5) can be optionally replaced with a nitrogen atom, and with the proviso that an adjacent pair of optional substituents can be bonded to form a cyclic structure.

2. A luminescence device according to Claim 1, wherein a partial structure ML'n in said formula (1) is represented by said formula (3).

- 3. A luminescence device according to Claim 1, wherein a partial structure ML'n in said formula (1) is represented by said formula (4).
- 4. A luminescence device according to Claim 1, wherein n in said formula (1) is 0.
- 5. A luminescence device according to any one of Claims 1 to 4, wherein the cyclic group B in said formula (1) comprises an isoquinoline skeleton.
- 6. A luminescence device according to Claim 5, wherein said isoquinoline skeleton is bonded to the cyclic group A at its 1-position.
- 7. A luminescence device according to any one of Claims 1 to 6, wherein the organic layer including said metal coordination compound comprises an electric-field luminescence device between a pair of oppositely disposed electrodes comprising a transparent electrode (anode) and a metal electrode (cathode) which are supplied with a voltage to cause luminescence.
- 8. A display apparatus, comprising a luminescence device as defined in any one of Claims 1

to 7, as a display device.

[Detailed Description of the Invention]
[Field]

The present invention relates to a luminescence device using an organic compound. More particularly, the invention relates to an organic electroluminescence device (also called an organic EL device) by use of a metal coordination compound represented by formula (1) appearing hereinafter, as a luminescence material.

### [Prior Art]

An extensive study for an organic EL device formation as a luminescence device of a high-speed responsiveness and a high efficiency, has been conducted. The basic structure of the organic EL device is illustrated in Figures 1A and 1B (for example, Macromol. Symp. 125, 1 - 48 (1997)).

As described in Figure 1, an organic EL device generally comprises, on a transparent substrate 15, a transparent electrode 14, a plurality of organic film layers and a metal electrode 11 formed so as to cover the organic layers.

Referring to Figure 1A, the organic layer comprises a luminescence layer 12 and a hole-transporting layer 13. The transparent electrode 14 may comprises ITO or the like, having a large work

function so as to facilitate hole injection from the transparent electrode 14 to the hole-transporting layer 13. The metal electrode 11 comprises a metal material having a small work function, such as aluminum, magnesium or alloys of these elements, so as to facilitate electron injection into the organic luminescence device. The film having the thickness of 50 to 200 nm is used for the electrodes.

In the luminescence layer 12, aluminum quinolinol complex (inclusive of Alq3 shown in the formula (3) as a representative example) having an electron-transporting characteristic and a luminescence characteristic, are used for example. In a hole-transporting layer, a material having an electron-donative property, such as a triphenyldiamine derivative (inclusive of  $\alpha$ -NPD shown in the formula (3) as a representative example), is used for example.

A device organized above exhibits a currentrectifying characteristic, and when an electric field
is applied between the metal electrode 11 as a
cathode and the transparent electrode 14 as an anode,
electrons are injected from the metal electrode 11
into the luminescence layer 12, and holes are injected
from the transparent electrode 15.

The injected holes and electrons are recombined in the luminescence layer 12 to form

excitons, which cause luminescence. In this instance, the hole-transporting layer 13 functions as an electron-blocking layer to increase the recombination efficiency at the boundary between the luminescence layer layer 12 and the hole-transporting layer 13, thereby providing an enhanced luminescence efficiency.

Further, in the structure of Figure 1(b), an electron-transporting layer 16 is disposed between the metal electrode 11 and the luminescence layer 12 in Figure 1(a). As a result, the luminescence function is separated from the functions of election transportation and hole transportation to provide a structure exhibiting more effective carrier blocking, thus increasing the luminescence efficiency. The electron-transporting layer 16, may comprise, e.g., an oxadiazole derivative.

In ordinary organic EL devices, fluorescence caused during a transition of luminescent center molecule form a single excited state to a ground state is used as luminescence. On the other hand, not the above fluorescence (luminescence) via singlet exciton, phosphorescence (luminescence) via triplet exciton has been studied for use in organic EL devices as described in, e.g., "Improved energy transfer in electrophosphorescent device" (D.F. O'drien et al., Applied Physics Letters. Vol. 74, No. 3, pp. 442 - 444 (1999)) and "Very high-efficiency green organic light-

emitting devices based on electrophosphorescence" (M.A. Baldo et al., Applied Physics Letters, Vol. 75, No. 1, pp. 4 - 6 (1999)).

The EL devices shown in these documents may generally have a sectional structure shown in Figure 1C. Referring to Figure 1C, four organic layers including a hole transfer layer 13, a luminescence layer 12, and exciton diffusion-prevention layer 17, and an electron transport layer 16 are successively formed in this order on the transparent electrode (anode) 14. The materials used therein include carrier-transporting materials and phosphorescent materials which is represented in KA-3, of which the names and structures are shown below together with their abbreviations.

Alq3: aluminum-quinolinol complex,

 $\alpha\text{-NPD:}\quad N4\,, N4\,'\text{-di-naphthalene-1-yl-N4}\,, N4\,'\text{-}$   $diphenyl\text{-biphenyl-4}\,, 4\,'\text{-diamine}\,,$ 

CBP: 4,4'-N,N'-dicarbazole-biphenyl,

BCP: 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline.

PtOEP: platinum-octaethyl porphine complex, and

Ir(ppy)3: iridium-phenylpyridine complex.

The above-mentioned Articles 1 and 2 both have reported structures, as exhibiting a high efficiency, including a hole-transporting layer 13 comprising a-NPD, an electron-transporting layer 16 comprising Alq3, an exciton diffusion-preventing layer 17 comprising BCP, and a luminescence layer 12 comprising CBP as a host and ca. 6 % of PtOEP or

Ir(ppy)<sub>3</sub>

 $Ir(ppy)_3$  as a phosphorescent material dispersed in mixture therein.

Such a phosphorescent material is particularly noted at present because it is expected to provide a high luminescence efficiency in principle for the following reasons. More specifically, excitons formed by carrier recombination comprise singlet excitons and triplet excitons in a probability ratio of 1:3. Conventional organic EL devices have utilized fluorescence caused by the transition of a singlet exciton to a ground state, of which the luminescence efficiency is limited to at most 25 %. On the other hand, if phosphorescence generated from triplet excitons is utilized, an efficiency of at least three times is expected, and even an efficiency of 100 %, i.e., four times, can be expected in principle, if a transition owing to intersystem crossing from a singlet state having a higher energy to a triplet state is taken into account.

The use of phosphorescence based on transition from the triplet excited state has also been proposed in, e.g., Japanese Laid-Open Patent Application No. HEI-11-329739 (organic EL device and manufacturing method for organic EL device), Japanese Laid-Open Patent Application No. HEI-11-256148 (luminescence material and organic EL device using the luminescence material) and HEI-8-319482 (organic

electroluminescence device).

[Problems to be Solved]

However, the above-mentioned organic EL devices utilizing phosphorescence have accompanied with a problem of the deterioration of luminescence efficiency and device stability. The reason for luminescent deterioration has been clarified as yet but may be attributable to such a phenomenon that the life of triplet exciton is generally longer than that of single exciton by at least three digits, so that molecule is placed in a higher-energy state for a long period to cause reaction with ambient substance, formation of exciplex or excimer, change in minute molecular structure, structural change of ambient substance or the like.

A luminescent center material used in a phosphorescent luminescence device is desired to exhibit high luminescence efficiency and show a high stability.

Accordingly, it is a principal object of the present invention to provide a luminescence device which exhibits a high luminescence efficiency and retains a high luminance for a long period, and having a luminescence wavelength of long-wavelength region, and a display device using the luminescence device.

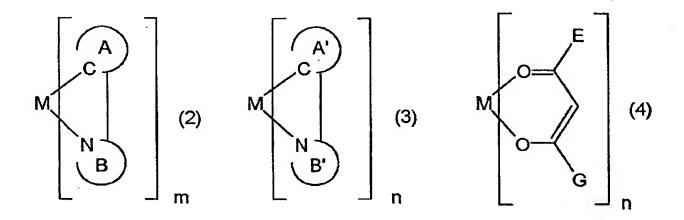
[Means for Solving the Problems]

More specifically, in accordance with a

principal aspect of the present invention, there is provided a luminescence device having a layer containing a metal coordination compound, which is represented by formula (1) below:

$$MLmL'n$$
 (1)

wherein M is a metal atom of Ir, Pt, Rh or Pd; L and L' are mutually different bidentate ligands; m is 1, 2 or 3 and n is 0, 1 or 2 with the proviso that m+n is 2 or 3; a partial structure MLm is represented by formula (2) shown below and a partial structure ML'n is represented by formula (3) or (4) shown below:



N and C are independently a nitrogen atom and a carbon atom; A and A' are each cyclic group capable of having an optional substituent, including a carbon atom and bonded to the metal atom M via the carbon atom; B and B' are each cyclic group capable of having an optional substituent, including a nitrogen atom and bonded to the metal atom M via the nitrogen atom; {(the optional substituent of the cyclic groups is selected from a halogen atom, cyano group, a nitro

group, a trialkylsilyl group (of which the alkyl groups are independently a linear or branched alkyl group having 1 to 8 carbon atoms), a linear or branched alkyl group having 1 to 20 carbon atoms (of which the alkyl group can include one or nonneighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH-  $\,$ or -C = C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom); or an aromatic group capable of having an optional substituent that is a halogen atom, a cyano atom, a nitro atom, a linear or branched alkyl group having 1 to 20 carbon atoms (of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C=C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom) }.

The cyclic groups A and B are bonded to each other via a covalent bond and the cyclic group A' and B' are bonded to each other via a covalent bond.

E and G are independently a linear or branched alkyl group having 1 to 20 carbon atoms (of which the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom), or an aromatic cyclic group capable of having an optional substituent {(that is a halogen atom, a cyano atom, a

nitro atom, a trialkylsilyl group (of which the alkyl groups are independently a linear or branched alkyl group having 1 - 8 carbon atoms), a linear or branched alkyl group having 1 to 20 carbon atoms (of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C≡C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom)}.

At least one of the optional substituents of the cyclic groups, and the cyclic groups B and B' include an aromatic cyclic group capable of having an optional substituent represented by the following formula (5):

<KA-5>

The optional substituent of the aromatic cyclic group is selected from a halogen atom, cyano group, a nitro group, a trialkylsilyl group of which the alkyl groups are independently a linear or branched alkyl group having 1 to 8 carbon atoms, a linear or branched alkyl group having 1 to 20 carbon atoms of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH-

or -C≡C-; and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom, or an aromatic group capable of having an optional substituent (that is a halogen atom, a cyano atom, a nitro atom, a linear or branched alkyl group having 1 to 20 carbon atoms of which the alkyl group can include one or non-neighboring two or more methylene groups that can be replaced with -O-, -S-, -CO-, -CO-O-, -O-CO-, -CH=CH- or -C $\equiv$ C-, and the alkyl group can include a hydrogen atom that can be optionally replaced with a fluorine atom) with the proviso that one or two CH groups comprising the aromatic cyclic group capable of having a substituent in the formula (5) can be optionally replaced with a nitrogen atom, and with the proviso that an adjacent pair of optional substituents can be bonded to form a cyclic structure.

A luminescence device according to the present invention may be preferable that a partial structure ML'n is represented by the formula (3) in the formula (1). A luminescence device according to the present invention may be preferable that a partial structure ML'n in the formula (1) is represented by the formula (4) in the formula (1). A luminescence device according to the present invention may be preferable that n in the formula (1).

A luminescence device according to the

present invention may be preferable that the cyclic group B is the isoquinoline skeleton in the formula (1), further preferable that the isoquinolyl skeleton is bonded to the cyclic group A at its 1-position.

A luminescence device may preferably that the organic layer comprising the metal coordination compound comprises an electric-field luminescence device between a pair of oppositely disposed electrodes comprising a transparent electrode (anode) and a metal electrode (cathode) which are supplied with a voltage to cause luminescence.

In accordance with a further aspect of the present invention, there is provided a display apparatus, comprising a luminescence device described above, as a display device.

[Description of the Preferred Embodiments]

In the case where the luminescence layer comprises a host material having a carrier-transporting function and a phosphorescent guest material, a process of phosphorescence via triplet excitons may include unit processes as follows:

- 1. transportation of electrons and holes within a luminescence layer,
  - 2. formation of host excitons,
- 3. excitation energy transfer between host molecules,
  - 4. excitation energy transfer from the host to

the guest,

- 5. formation of guest triplet excitons, and
- 6. transition of the guest triplet excitons to the ground state and phosphorescence.

Desirable energy transfer in each unit process and luminescence are caused in competition with various energy deactivation processes.

Needless to say, a luminescence efficiency of an EL device is increased by increasing the luminescence quantum yield of a luminescence center material. In addition thereto, an efficiency energy transfer between host material molecules and/or between host material molecule and guest material molecule is also an important factor. Further, the above-described luminescent deterioration in energized state may presumably relate to the luminescent center material per se or an environmental change thereof by its ambient molecular structure.

As a result of various studies of inventors, it has been found that an organic EL device using the metal coordination compound of the formula (1) as a principal luminescence material exhibits high-efficiency luminescence and has a luminescence wavelength of long-wavelength region, retains high luminance for a long period.

It is preferable that a partial structure ML'n of metal coordination compounds represented in

the formula (1) is represented by the formula (3), that a partial structure ML'n is represented by the formula (4), and that n is 0 in the formula (1). Further, it is preferable that the cyclic group B is the isoquinolyl skeleton in the formula (1) further preferable that the isoquinolyl skeleton is bonded to the cyclic group A at its 1-position.

The metal coordination compound used in the present invention emits phosphorescence, and its lowest excited state is believed to be an MLCT\* (metal-to-ligand charge transfer) excited state in a triplet state, and phosphorescence is caused at the time of transition from such a state to the ground state.

The luminescence material exhibited high phosphorescence quantum yields of 0.15 to 0.9 and short phosphorescence lives of 1 to 30 µsec. A short phosphorescence life becomes a condition for causing little energy deactivation and exhibiting an enhanced luminescence efficiency. More specifically, if the phosphorescence life is long, the number of triplet state molecules maintained for luminescence is increased, and the deactivation process is liable to occur, thus resulting in a lower luminescence efficiency particularly at the time of a high-current density. The material of the present invention is suitable as a luminescence material for an EL device

having a high phosphorescence quantum yield and a short phosphorescence life. Further, by introducing an isoquinoline skeleton in a metal coordination compound having a structure of a type represented by the above formula (1), the luminescence wavelength can be adjusted, and it has been found that the metal coordination compound of the present invention wherein the isoquinoline skeleton is bonded to the cyclic group A at its position-1, is unexpectedly advantageous for increasing the luminescence wavelength. From this fact, a metal coordination compound of the present invention is suited as a luminescence material for an EL device.

In an actual current conduction test, the luminescence material of the present invention, i.e., a metal coordination compound having a ligand comprising an isoquinoline skeleton boned to a cyclic group A at its 1-position, showed a high stability.

It is believed that the ligand of the present invention, as a result of introduction of isoquinoline skeleton, has a rigid molecular structure, so as to suppress the formation of an excitation-associated molecule resulting in thermal deactivation, thus suppressing energy deactivation due to molecular movement. Further, it is also believed that extinction processes are reduced to result in an improve device performance. Accordingly, a

luminescence material having a luminescence wavelength of long-wavelength region (red luminescence) and a high chemical stability as well as a high luminescence efficiency has not been realized heretofore but can be realized by the luminescence material of the present invention.

The luminescence device may preferably that the organic layer including the metal coordination compound represented by the formula (1) comprises an electric-field luminescence device between a pair of oppositely disposed electrodes comprising a transparent electrode (anode) and a metal electrode (cathode) which are supplied with a voltage to cause luminescence.

The luminescence device having a high efficiency of the present invention is applicable to the product which requires for energy saving and high luminosity. As an example of application, the light source of a display apparatus and an illumination apparatus or printer, the back light of a liquid crystal display apparatus or the like, can be considered. As for a display apparatus, energy saving, and high visibility and a light flat panel display are provided. As for a light source of a printer, the laser light source of the laser beam printer used widely can be transposed to the luminescence device. The device which can carry out

an address independently is disposed on an array, and image formation is performed by performing a desired exposure process to the photosensitive drum. By using the device of the present invention, the volume of the apparatus can be decreased sufficiently. The energy saving effect according to the present invention is expected regarding the illumination apparatus and back light.

For the application to a display, a drive system using a thin-film transistor (abbreviated as TFT) drive circuit according to an active matrix-scheme, may be used.

Hereinbelow, an embodiment of using a device of the present invention in combination with an active matrix substrate is briefly described with reference to Figures 4 to 6.

Figure 4 illustrates an embodiment of panel structure comprising an EL device and drive means. The panel is provided with a scanning signal driver, a data signal driver and a current supply source which are connected to gate selection lines, data signal lines and current supply lines, respectively. At each intersection of the gate selection lines and the data signal lines, a display pixel electrode is disposed. The scanning signal drive sequentially selects the gate selection lines G1, G2, G3 ... Gn, and in synchronism herewith, picture signals are supplied

from the data signal driver to display a printer.

Next, the operation of a pixel electrode will be described. In the pixel electrode, when the selection signal is supplied to a gate selection line, TFT1 is set to being turned on, and a picture signal is supplied to Cadd, and then, the gate potential of TFT2 is determined. The current is supplied to the EL element through the current supply line according to the gate potential of TFT2. The gate potential of TFT2 is retained Cadd until the next scan is performed for TFT1, so that the EL element is held at Cadd until next scanning selection of the TFT1 is carried out, and therefore, the luminescence can be emitted during one frame period.

Figure 6 is a schematic view of an example of a sectional structure of TFT substrate to be used for the present invention. A p-Si layer is provided on a glass layer. The impurities respectively required for the regions of channel, a drain, and a source are doped. The gate electrode is provided through the gate insulator, and simultaneously, the dorain electrode and source electrode connected to the drain region and the source region are formed. The insulator layer and the ITO electrode as the pixel electrode are accumulated on the drain electrode and the source electrode. The ITO electrode is connected to the drain electrode through a contact hole.

The present invention is not limited to the switching device, however, it may be also applicable easily to a substrate of a single crystal silicone, a MIM device or a-Si type element or the like.

A multiplayer or single layer organic EL layer and cathode layer is accumulated one by one on the ITO electrode, and an organic display panel can be provided. By driving a display panel including a luminescence layer comprising a luminescence material of the present invention, it becomes possible to provide a display which exhibits a good picture quality and is stable even for a long period display.

Some synthetic paths for providing a metal coordination compound represented by the above-mentioned formula (1) are illustrated below with reference to an iridium coordination compound, for example.

Synthetic of ligand L (Reference; Kevin R, et al., Org., Lett., 1999, 1, 553 - 556)

<KA-6>

<KA-7>

<Ka-8>

Synthetic of iridium coordination compound

$$Ir(CH_3COCHCOCH_3)_3 \xrightarrow{3 \times L} Ir(L)_3$$
or
$$IrCl_3 \xrightarrow{2 \times L} [Ir(L)_2Cl]_2 \xrightarrow{Lor L'} Ir(L)_3 \text{ or } Ir(L)_2L'$$

Some specific structural examples of metal coordination compounds used in the present invention are shown in Table 1 to Table 25 appearing hereinafter, which are however only representative examples and are not exhaustive. Ph to Py2 used for Table 1 to Table 25 represent partial structures shown below.

Table 1

_		_	-	_	_		<del></del>	-			_									
B'-R6	1	j	1	i,	1	f	l	1	l	<b>l</b>	l	1	1	-		ı	1	1	I	1
B'-R5		ŀ	1	1	_	1	1	1	ı	1	-	1	1	-	ŧ	1		_	ı	•
B'-R4	1	1	ı	ì	١	•	t	ł	ı	1	-	-	1	ı		€ .		-	1	ı
B'-R3	ı	ŧ	-	ı	1	1	_	l		ı	-	-	1	-	1	E	ı	_	l	1
B-R6	I	H	Н	¥	I	Н	Ή	I	r	I	I	Н	I	I	ĸ	Ξ	Ŧ	I	π	Ή
B-RS	Ξ	I	I	I	CF3	I	X	π	I	±	I	H	×	Н	I	×	Ŧ	Ŧ	Ŧ	Ή
B-R4	I	I	Н	£	Ŧ	CF3	H	Ξ	I	×	Ŧ	H	3:	Ι	Ŧ	æ	I	Ξ	£	·
B-R3	I	I	Ŧ	I	I	Ι	π	I	Ξ	æ	Ξ	Ŧ	π.	Ξ	I	Ŧ	Ŧ	Н	I	Ξ
A'-R2	-	ı	,	1	1	1	ı	1	1	I	1	ı	1	ı	1	t	1	ı	ı	J
A'-R1		j	1	1	1		1	ı	ı	1	ı	,	1	<b>I</b>	l	ı		1	i	
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Table 2

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B'-R4	_	-	•	1	1	•	•	1	1	1	1	<b>\$</b>	1	1	1	ı		_ = _	ļ	_
B'-R3	1	-	1	1	ŧ	ſ	ı	ı	1	1	ı	l	ı	1	1	1	-		ı	-
B-R6	I	I	π	Τ	I	I	I	Ι	CF3	I	н	x	π	r	I	I	Ŧ	H	H	Ξ
B-R5	Н	н	I	Н	Н	н	CF3	Ή	H	I	π.	I	H	I	π	8	×	н	£	Ξ
B-R4	H	I	Ę	Н	Н	CF3	Н	H	Н	. H	I	Ξ	I	I	3	I	윤	ĸ	×	×
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Table 3

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B'-R3	•	,	,	,			•	1	,		1	-	1	,		1	1	1		ı	ı	1	1	١	
B-R6	I	=	3	3	-	-	I	I	I		C.	H	Ξ	3	-	I	I	Ι		I	τ	I	I	1	
8-R5	=	-	-	- -		Ξ	I	CF3	=		π	Ŧ	æ		E	Ŧ	CF3	I		I	×	Ŧ	I	3	E.
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Table 4

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B'-R3	-	_	ŀ	i	1	,	_	ı	,	-	ı	1	-		,		1	-		-
B-R6	н	X.	I	I	×	I	H	π	I	I	x	Ι	Ξ	I	I	F	H	н	Н	I
B-R3	Ŧ	I	Ŧ	×	×	Ŧ	Н	x	Ξ	£	Ι	8	Ξ	CF3	Ŧ	н	H	F	н	포
B-R4	Н	н	π	Ξ	×	Ŧ	Н	Æ	£	×	8	x	CF3	Ξ	u.	H	H	Н	Н	Ξ
23 <del>4</del> -8	I	Ή	I	I	r	Ŧ	H	Ŧ	I	I	н	Ι	±	±	Ŧ	Н	F	Ŧ	Н	I
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A'-R1	•	_	1	1	ļ	1	1	l	i	-	1	ı	,	1	1	1	`	-	_	1
A-R2	CF3	Ξ	\$	I	I	H	OCHB	x	I	H	π	I	Ξ	I	Ŧ	Н	H	I	I	I
A-R1	н	X	I	occ	\$	OCH3	x	8	H	Н	Ή	x	Ξ	Ξ	I	Н	Ξ	X	осн2сн2	Ŧ
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Table 5

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B'-R4	1	1	ı	_	ı	,	ı	-	ı	,		1	1	-	ı	ł.	1	ı	1	-
B'-R3	,		1	_	1		,	1	\$	1	1	ı	1	1	-	. !	1	1	ı	
B-R8	I	I	Ξ	Н	I	Ŧ	H	H	I	I	H	I	Ξ	Ι	I	x	x	I	· <b>x</b>	I
B-RS	I	H	Н	Ph	H	Ξ	Н	CF3	H	r	¥	· I	Ξ	CF3	Ŧ	I	π	I	x	Ξ
B-R4	I	Ŧ	£	H	Ŧ	Ŧ	CF3	Н	Ŧ	π	н	Ξ	£	н	CF3	Ŧ	Ŧ	x	I	Ξ
B-R3	Ξ	I	H	H	I	Ŧ	Ŧ	Ŧ	CF3	Ŧ	T	I	Ŧ	π	Ξ	x	Ŧ	Ŧ	I	Ŧ
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A-R2	Ŧ	æ	н	Ŧ	Ή	땑	H	x	Ħ	H	Ŧ	£	н	н	CH3	н	∞ .	x	$\varphi$	Ι
A-R1	Æ	Ŧ	ፎ	Ŧ	CH3	н	Ŧ	н	H	н	Æ	8	Æ	뚔	I	8	Ι	$\stackrel{\text{co}}{\sim}$	Ŧ	SE3
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c	0		0		0	0	-	0		0	0	0	0	0	0	0	0	0	0	0
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Table 6

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B'-R3	ı	1	Ē.	1	ı	ı	ı	ı	1	~	I	1	_	)		•	-		1	1
B-R6	H	н	I	I	x	x	I	Ξ	I	Н	I	Ι	H	н	I	<u>.</u>	H	н	H	I
B-R5	Н	H	π	I	Œ	r	Ξ	I	Ξ	Æ	Ξ	8	Ŧ	ಆ೦	H	Н	н	F	¥	Ξ
B-R4	Ŧ	I	π	π	I	I	Ξ	£	Æ	H	8	π.	CF3	I	F	Ξ	Н	Ŧ	Ŧ	Ξ
B-R3	Η	I	I	I	Ŧ	×	Ξ	I	Ξ	н	Ι	I	I	I	I	H	H.	н	H	Ŧ
A'-R2	_	ı	ı	1	ı	,	-	1	1	-	-	1		-	1	-	3	ī	i	1
A'-R1	-	1	ſ	1	1	ı	-	ı	ı		1	ı	_	_	-	-		-	_	1
A-R2	CF3	Ι	8	H	x	Ξ	OCH3	x	Ξ	Ŧ	I	I	I	H	Н	H	Ŧ	Ŧ	H	Ξ
A-R1	H	\$	x	2000	\$	OCH3	Н	8	Ξ	Ξ	Ι	Ξ	x	I	ж	H	×	H	SH2SH2	Ξ
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Table 7

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B-84	-	I		£	I	CF3	Ŧ	×	Ξ	I		=	=	I	Ξ	Ξ	1 2	=	E	=	đ,	ð	Ŧ
B-R3	1	r	-	F	I	H	×	F	x	I		=	<b>→</b>	I	Ξ	Ξ	=		=	Ŧ	I	-	I
A'-R2	,			,		'	ı	1	1	1		•	,	1	1	-	,	+		1	ŀ	<b>†</b> ,	
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Table 8

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M   M   M   M   M   M   M   M   M   M		B'-R4	1	1	1	1	1		1		†	1		1	-	,	•							1	1
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Table 11

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B-F3	I	Ξ	I	I	Ξ	I	=	E	Ŧ	=	Ξ	Ξ	Ŧ	Ξ	Ξ	I	=	CF3	±	Ξ
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Table 12

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Table 13

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B-R3	I	H	I	x	H	I	Ŧ	I	I	Ŧ	×	H	Ξ	×	Ŧ	I	I	Ξ	I	¥
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Table 14

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8-84	Ŧ	Ξ	I	Ξ	I	Ŧ	Ξ	H	I	=	Ŧ	I	I	=	Ξ	I	Ŧ	=	Ŧ	Ξ
B-R3	I	Ξ	×	×	Ŧ	Ŧ	Ξ	н	Ŧ	Ŧ	I	Ŧ	I	Ξ	Ξ	Ξ	H	Ŧ	Ξ	Ξ
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Table 23

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Table 24

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Handra   March   M	A-F2	Ξ	CH3	딵	I	F	Н	Н	н	н	н	Н	H	CH3	H	н	H	ଫ	Ξ	ű.	I
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Table 25

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#### [Examples]

Hereinafter, the present invention will be described more specifically based on Examples.

<Examples 1 - 2>

The structure including four organic layers as shown in Figure 1 was prepared as the structure of the device. The opposing electrode area is made into  $3 \text{ mm}^2$  by patterning a 100 nm-thick film (transparent substrate 14) of ITO on a glass substrate (transparent substrate 15). On the ITO-formed substrate, three organic layers and two metal electrode layers shown below were successively formed by vacuum (vapor) deposition using resistance heating in a vacuum chamber  $(10^{-4} \text{ Pa})$ .

Organic layer 1 (hole transport layer 13)
(40 nm): a-NPD

Organic layer 2 (luminescence layer 12) (30 nm): co-deposited film of CBP: metal complex (metal coordination compound) (8 wt. %)

Organic layer 3 (electron transport layer 16) (30 nm): Alq3

Metal electrode layer 1 (metal electrode 11)
(15 nm): Al-Li alloy (Li = 1.8 wt. %)

Metal electrode layer 2 (metal electrode 11) (100 nm): Al

As the ligands, Example Compound No. 1, 170 was used.

The performances of the thus-obtained EL devices were measured by using a micro-current meter ("4140B", made by Hewlett-Packard Corp.) for a current-voltage characteristic and "BM7" (made by Topcon K.K.) for an emission luminance. The devices using the respective coordination compounds respectively exhibited a good rectifying characteristic.

At an applied voltage of 12 volts, the EL devices exhibited luminescence as follows:

Device of Example 1 (Compound No. 1):

 $8000 \text{ cd/cm}^2$ 

Device of Example 2 (Compound No. 28):

 $1000 \text{ cm/m}^2$ 

The above-mentioned luminescence of the EL device almost coincide with the photoluminescence measured by dissolving the luminescence material used in this embodiment in toluene, whereby it was confirmed that the luminescence of the EL device were emitted from the above-mentioned luminescence material.

<Examples 3 ~ 7, Comparative Example 1>

Luminescence devices were prepared in the same manner as in Examples 1 and 2 except for using luminescence materials (Example Compounds) shown in Table 26 below. In Comparative Example 1, the abovementioned Ir(ppy)<sub>3</sub> represented by Ka-3 was used as a representative of conventional luminescence material.

The measurement results of a current conduction durability test for each luminescence devices are shown in Table 26 and the Example materials exhibited a luminance half-attenuation period which was clearly longer than the conventional luminescence material, thus providing a device having a high durability attributable to the material of the present invention.

Table 26

	Luminescence material No.	Luminance half-attenuation period (hours)
Ex. 3	1	700
Ex. 4	51	490
Ex. 5	91	800
Ex. 6	120	850
Comp. 1	170	900
Comp. 2	Ir(ppy) <sub>3</sub>	350

#### <Example 8>

A simple matrix type organic EL device as shown in Figure 2 was prepared in the following manner.

On a glass substrate 21 measuring 75 mm-length, 75 mm-width and 1.1 mm-thickness, a ca. 100 nm-thick ITO film was formed by sputtering and

patterned into 100 lines of 100 µm-wide transparent electrodes 22 (anode side) with a spacing of 40 µm as simple matrix electrodes. Then, formed layers of identical organic materials were found under identical conditions as in Example 2 to form an organic compound layer 23.

Then, 100 lines of 100 µm-wide Al electrodes 24 were formed with a spacing of 40 µm by mask vacuum deposition so as to be perpendicular to the transparent electrodes 22 by vacuum deposition at a vacuum of  $2.7 \times 10^{-3}$  Pa ( $2 \times 10^{-5}$  Torr). The metal electrodes (cathode) 24 were formed as a lamination of 10 nm-thick layer of Al/Li alloy (Li: 1.3 wt. %) and then 150 nm-thick layer of Al.

The thus-obtained 100x100-simple matrix-type organic EL device was subjected to a simple matrix drive in a glove box filled with nitrogen at voltages of 7 volts to 13 volts by using a scanning signal of 10 volts and data signals of ±3 volts. As a result of an interlaced drive at a frame efficiency of 30 Hz, respectively, luminescence images could be confirmed. <Example 9> (Synthesis of Example Compound No. 231) <Ka-10>

$$\bigcap_{N \to 0} \bigcap_{N \to \infty} \bigcap_{N$$

69.3 g (448 mmol) of isoquinoline N-oxide (made by Tokyo Kasei) and 225 ml of chloroform were placed and dissolved in a 1 liter-three-necked flask, and under stirring and cooling with ice, 219.6 g (1432 mmol) of phosphorus oxychloride was gradually added dropwise thereto while the internal temperature was held at 15 - 20  $^{\rm O}$ C. Thereafter, the temperature was raised, and reflux under stirring was performed for 3 The reaction product was cooled by standing to room temperature and poured into iced water. After extraction with ethyl acetate, the organic layer washed with water until neutrality, and the solvent was removed under a reduced pressure to provide a dry solid, which was then purified by silica gel column chromatography (eluent: chloroform/hexane = 5/1) to obtain 35.5 g (yield: 44.9 %) of 1-chloroisoquinoline white crystal.

<Ka-11>

In a 100 ml-three-necked flask, 3.04 g (24.9 mmole) of phenylboronic acid, 4.0 g of (25.0 mmole) of 1-chloroisoquinoline, 25 ml of toluene, 12.5 ml of ethanol and 25 ml of 2M-sodium carbonate aqueous solution were placed and stirred at room temperature

under nitrogen stream, and 0.98 g (0.85 mmole) of tetrakis(triphenylphosphine)palladium (0) was added Thereafter, reflux under stirring was thereto. performed for 8 hours under nitrogen stream. completion of the reaction, the reaction product was cooled and extracted by addition of cold water and The organic layer was washed with saline toluene. water and dried on magnesium sulfate, followed by removal of the solvent under a reduced pressure to The residue was purified by silica provide dry solid. gel column chromatography (eluent: chloroform/methanol = 10/1) to obtain 2.20 g (yield = 43.0 %) of 1phenylisoquinoline.

<Ka-12>

In a 100 ml-four-necked flask, 50 ml of glycerol was place and heated at 130 - 140  $^{\rm O}{\rm C}$  under stirring and bubbling with nitrogen for 2 hours. Then, the glycerol was cooled by standing down to 100  $^{\rm O}{\rm C}$ , and 1.03 g (5.02 mmole) if 1-phenylisoquinoline and 0.50 g (1.03 mmole) of iridium (III) acetyl-

acetonate were added, followed by 7 hours of heating around  $\pm 210^{\circ}$ C under stirring and nitrogen stream. The reaction product was cooled to room temperature and injected into 300 ml of 1N-hydrochloric acid to form a precipitate, which was filtered out and washed with water. The precipitate was purified by silica gel column chromatography with chloroform as the eluent to obtain 0.22 g (yield = 26.8 %) of red powdery tris(1-phenylisoquinoline-C2,N)iridium (III). A chloroform solution of the compound exhibited a luminescence spectrum showing  $\lambda$ max = 619 nm.

An EL device was prepared in the same manner as in Example 1 except for using the compound instead of Compound No. 1 and was confirmed to emit red luminescence showing  $\lambda$ max = 620 nm under voltage application.

<Example 10> (Synthesis of Example Compound No. 417) <Ka-13>

$$B(OH)_2$$
  $CI$ 

In a 100 ml-three-necked flask, 2.91 g (12.2 mmole) of 9,9-dimethylfluorene-2-boronic acid, 2.00 g (12.2 mmole) of 1-chloroisoquinoline, 10 ml of

toluene, 5 ml of ethanol and 10 ml of 2M-sodium carbonate aqueous solution were placed and stirred at room temperature under nitrogen stream, and 0.44 g (0.38 mmole) of tetrakis(triphenylphosphine)palladium (0) was added thereto. Thereafter, reflux under stirring was performed for 5 hours under nitrogen After completion of the reaction, the stream. reaction product was cooled and extracted by addition of cold water and toluene. The organic layer was washed with saline water and dried on magnesium sulfate, followed by removal of the solvent under a reduced pressure to provide dry solid. The residue was purified by silica gel column chromatography (eluent: toluene/ethyl acetate = 50/1) to obtain 2.13 g (yield = 54.2 %) of 1-(9,9-dimethylfluorene-2yl)isoquinoline.

<Ka-14>

**1** 

In a 100 ml-four-necked flask, 50 ml of glycerol was placed and heated at 130 - 140 °C under stirring and bubbling with nitrogen for 2 hours. Then, the glycerol was cooled by standing down to 100  $^{\mathrm{O}}\mathrm{C}$ , and 1.61 g (5.01 mmole) of 1-(9,9dimethylfluorene-2-yl)isoquinoline and 0.50 g (1.02 mmole) of iridium (III) acetylacetonate were added, followed by 8 hours of reflux under stirring and nitrogen stream. The reaction product was cooled to room temperature and injected into 600 ml of 1Nhydrochloric acid to form a precipitate, which was filtered out and washed with water. The precipitate was purified by silica gel column chromatography with chloroform as the eluent to obtain 0.38 g (yield = 32.3 %) of red powdery tris[1-(9,9-dimethylfluorene-2yl)isoquinoline-C<sup>3</sup>,N]iridium (III). A toluene solution of the compound exhibited a luminescence spectrum showing  $\lambda max = 648 \text{ nm}$ .

An EL device was prepared in the same manner as in Example 1 except for using the compound instead of Compound No. 1 and was confirmed to emit red luminescence showing  $\lambda$ max = 650 nm under voltage application.

<Example 11> (Synthesis of Example Compound No. 414)
<Ka-15>

In a 100 ml-three-necked flask, 4.45 g (25.0 mmole) of thianaphthene-2-boronic acid (made by Aldrich Chemical Co., Inc.), 4.09 g (25.0 mmole) of 1chloroisoquinoline, 25 ml of toluene, 12.5 ml of ethanol and 25 mol of 2M-sodium carbonate aqueous solution were placed and stirred at room temperature under nitrogen stream, and 0.98 g (0.85 mmole) of tetrakis(triphenylphosphine)palladium (0) was added Thereafter, reflux under stirring was performed for 8 hours under nitrogen stream. completion of the reaction, the reaction product was cooled and extracted by addition of cold water and The organic layer was washed with saline toluene. water and dried on magnesium sulfate, followed by removal of the solvent under a reduced pressure to provide dry solid. The residue was purified by silica gel column chromatography (eluent: chloroform) to obtain 4.20 g (yield = 64.3 %) of 1-(thianaphthene-2yl)isoquinoline.

<Ka-16>

In a 100 ml-four-necked flask, 50 ml of glycerol was placed and heated at 130 - 140  $^{\rm o}{\rm C}$  under stirring and bubbling with nitrogen for 2 hours. Then, the glycerol was cooled by standing to 100 °C. and 1.31 g (5.01 mmole) of 1-(thianaphthene-2-yl)isoquinoline, and 0.50 g (1.02 mmole) of iridium (III) acetylacetone, were added, followed by 5 hours of heating around 210  $^{\rm O}{\rm C}$  under stirring and nitrogen stream. The reaction product was cooled to room temperature and poured into 300 ml of 1N-hydrochloric acid to form a precipitate, which was then filtered out and washed with water. The precipitate was purified by silica gel column chromatography with chloroform as the eluent to obtain 0.25 g (yield = 25.2 %) of red powdery tris[1-(thianaphthene-2-yl)isoquinoline-C<sup>3</sup>,N]iridium (III). A toluene solution of the compound exhibited a luminescence spectrum showing  $\lambda \max = 686 \text{ nm}$ .

An EL device was prepared in the same manner as in Example 1 except for using the compound instead of Compound No. 1 and was confirmed to emit deep red luminescence under voltage application.

<Example 12> (Synthesis of Example Compound No. 413) <Ka-17>

In a 100 ml-three-necked flask, 2.56 g (20.0 mmole) of 2-thiophene-2-boronic acid (made by Aldrich Co.), 3.27 g (20.0 mmole) of 1-chloroisoquinoline, 18 ml of toluene, 9 ml of ethanol and 18 mol of 2M-sodium carbonate aqueous solution were placed and stirred at room temperature under nitrogen stream, and 0.72 g (0.62 mmole) of tetrakis(triphenylphosphine)palladium (0) was added thereto. Thereafter, reflux under stirring was performed for 9 hours under nitrogen After completion of the reaction, the reaction product was cooled and extracted by addition of cold water and toluene. The organic layer was washed with saline water and dried on magnesium sulfate, followed by removal of the solvent under a reduced pressure to provide dry solid. The residue was purified by silica gel column chromatography

(eluent: chloroform) to obtain 2.40 g (yield = 56.8 %)
of 1-(2-thienyl)isoquinoline.
<Ka-18>

In a 100 ml-four-necked flask, 50 ml of glycerol was placed and heated at 130 - 140  $^{\rm O}{\rm C}$  under stirring and bubbling with nitrogen for 2 hours. Then, the glycerol was cooled by standing to 100  $^{\rm o}$ C, and 1.05 g (4.97 mmole) of 1-(2-thienyl) isoquinoline, and 0.50 g (1.02 mmole) of iridium (III) acetylacetone, were added, followed by 8 hours of reflux under stirring and nitrogen stream. reaction product was cooled to room temperature and poured into 600 ml of 1N-hydrochloric acid to form a precipitate, which was then filtered out and washed The precipitate was purified by silica with water. gel column chromatography with chloroform as the eluent to obtain 0.38 g (yield = 45.2 %) of red powdery tris[1-(2-thienyl)isoquinoline-C<sup>3</sup>,N]iridium (III). A toluene solution of the compound exhibited a luminescence spectrum showing  $\lambda max = 642 \text{ nm}$ .

An EL device was prepared in the same manner as in Example 1 except for using the compound instead of Compound No. 1 and was confirmed to emit red luminescence showing  $\lambda = 640$  nm under voltage application.

### <Examples 13 - 15>

An EL device was prepared in the same manner as in Example 3 except for using the luminescence material represented in Table 27 as a metal coordination compound. A current conduction durability test was performed in the same manner as in Example 3. The measurement results are shown in Table 27. According to the measurement results, it was confirmed that when the isoquinolyl skeleton is bonded to the cyclic group A at its 1-position in the luminescence devices according to the present invention, a device having a high durability is provided.

Table 27

	Luminescence material No.	Luminance half-attenuation period (hours)
Ex. 13	231	1550
Ex. 14	413	1100
Ex. 15	417	1350

## [Advantageous Effect]

As described in foregoing, according to the present invention, a luminescence device of the present invention using, as a principle luminescence material, a metal coordination compound of the formula (1) is an excellent device which not only allows high luminescence efficiency at a high brightness (or luminance) for a long period while increasing the luminescence wavelength. Further, the display apparatus which exhibits an excellent display performance of the image by using the luminescence device of the present invention.

[Brief Description of the Drawings]

Figures 1A, 1B and 1C are schematic sectional views of an embodiment of a luminescence device according to the present invention, respectively.

Figure 2 illustrates a simple matrix-type organic EL device according to Example 8.

Figure 3 illustrates drive signals used in Example 8.

Figure 4 schematically illustrates a panel structure including an EL device and drive means.

Figure 5 is a schematic view of an example of a pixel electrode.

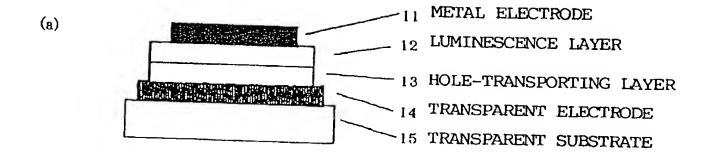
Figure 6 illustrates an embodiment of a sectional structure of TFT (thin-film transistor)

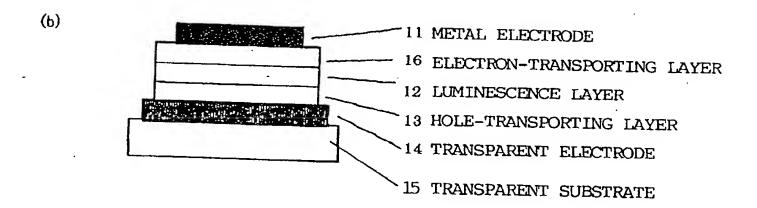
### substrate.

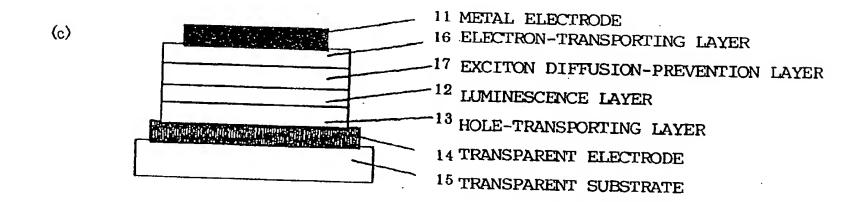
# [Reference Numerals]

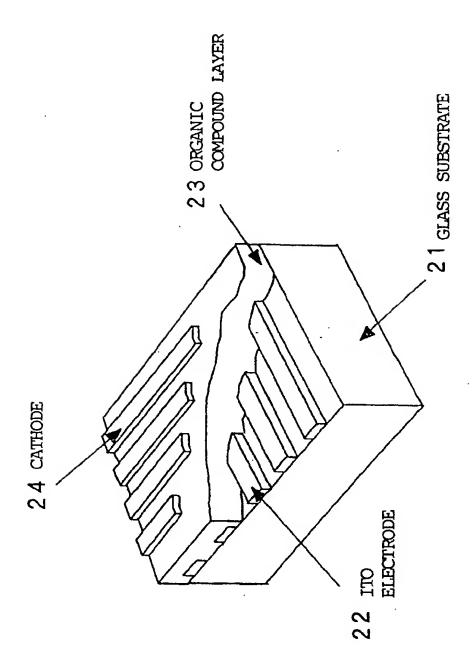
- 11: metal electrode
- 12: luminescence layer
- 13: hole transfer layer
- 14: transparent electrode
- 15: transparent substrate
- 16: electron transport layer
- 17: exciton diffusion-prevention layer
- 21: glass substrate
- 22: ITO electrode (transparent electrode)
- 23: organic compound layer
- 24: cathode

FIG. 1









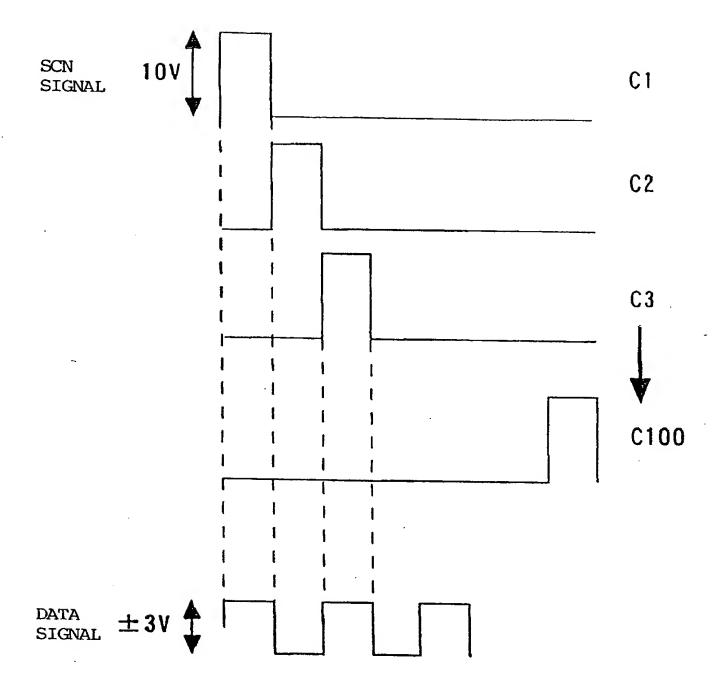


FIG. 4

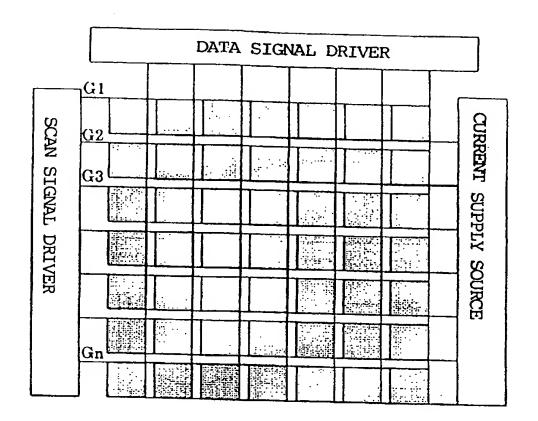
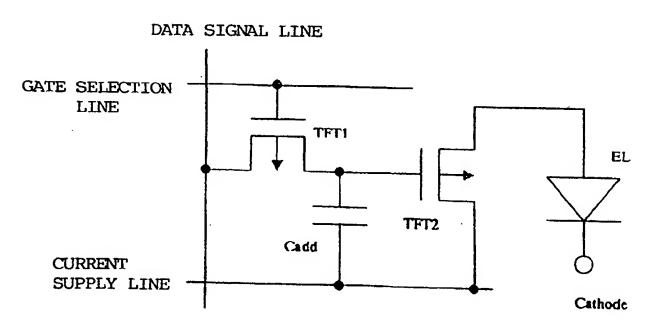
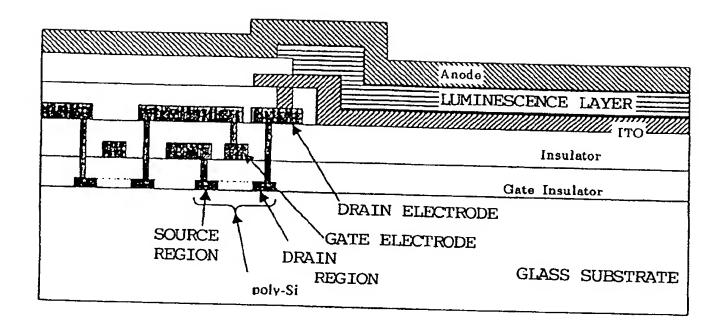


FIG. 5





## [Document]

Abstract

[Abstract]

[Object]

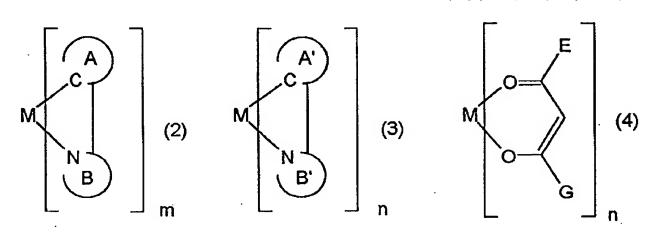
It is a principal object of the present invention to provide a luminescence device which exhibits a high luminescence efficiency and have a luminescence wavelength of long-wavelength region, and retains a high luminance for a long period.

[Means for Solving]

There is provided a luminescence device having a layer containing a metal coordination compound, which is represented by formula (1) below:

MLmL'n (1)

wherein M is a metal atom of Ir, Pt, Rh or Pd; L and L' are mutually different bidentate ligands; m is 1, 2 or 3 and n is 0, 1 or 2 with the proviso that m+n is 2 or 3; a partial structure MLm is represented by formula (2) shown below and a partial structure ML'n is represented by formula (3) or (4) shown below: <KA-1>





[Selected Figure]
Figure 1